# Organiskā ķīmija / Organic chemistry

81st International Scientific Conference of the University of Latvia 2023

# Report of Contributions

Contribution ID: **2** Type: **not specified**

# SYNTHETHIC PATHWAYS TOWARDS PURINE DERIVATIVE AS A POTENTIAL MOLECULAR SYSTEM FOR THE PHOTO-CATALYSIS

*Friday, 17 March 2023 11:00 (20 minutes)*

Target purine compound **2** was designed with an aim to be used as a potential system for photo-catalysis. For the synthesis of **2**, derivatization of *C*(6), *C*(8) and *N*(9) positions of 6-chloropurine (**1**) with **A**, **B** and **C** moieties is required. Several synthetic pathways were designed and have been tested. In the end, target compound **2** was obtained, using the combinations of S<sub>N</sub>Ar, S<sub>N</sub>2, CuAAC, C-C metal catalyzed coupling, alkylation and Mitsunobu reactions and these results will be discussed.

**Primary authors:** BURCEVS, Aleksejs (Riga Technical University); NOVOSJOLOVA, Irina

**Presenter:** BURCEVS, Aleksejs (Riga Technical University)

**Session Classification:** Organic chemistry session

Contribution ID: **3** Type: **not specified**

# EXPLORING THE REACTIVITY OF C(sp2)-H ACTIVATED AMINO ACID COBALT COMPLEXES: A FACILE ROUTE TOWARDS INDOLES

*Friday, 17 March 2023 09:00 (20 minutes)*

In the last few decades transition metal-catalyzed direct C-H bond functionalization has served as a valuable tool for the construction of complex molecules from more simple starting materials, mainly due to its atom- and step-economical nature. Nowadays, the field of third row transition metal catalyzed C-H functionalization is being extensively studied as a cheaper and attractive alternative to noble metal catalysts.
Our current work is dedicated to the development of cobalt-catalyzed picolinamide-directed C-H bond functionalization of amino acid derivatives. Starting from α,β-unsaturated amino acids 1 we were able to synthesize different C-H activated Co(III) complexes 2 in very good yields. Moreover, using N-fluorobenzenesulfonimide, indole 3 derivatives can be obtained.

**Primary author:** Mr ČIŽIKOVS, Aleksandrs (Latvian Institute of Organic Synthesis)

**Presenter:** Mr ČIŽIKOVS, Aleksandrs (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **4** Type: **not specified**

# COBALT-CATALYZED C(sp2)-H BOND ALLYLATION

*Friday, 17 March 2023 12:20 (20 minutes)*

In the last couple of decades, high-valent cobalt catalysis has been used as a valuable tool for C-H bond activation and functionalization.1 The use of cobalt(II) salt catalysts in combination with bidentate directing groups has proven to be an effective strategy for various C-H bond transformations.2,3 With cobalt being less expensive alternative to noble metals, it also displays unique reactivity and regioselectivity.4
Allyl- functional groups are important in organic synthesis as they open the door to many further modifications of the substrate. Employing cobalt catalyzed C-H bond allylation on amino acid derivatives 1, it is possible to utilize cheap reagents to obtain useful building blocks for other synthetic applications. Using optimization of cobalt catalysts, solvents, oxidants, additives and allylation reagents we were able to obtain diallylated phenylalanine derivative 2 in good yield.

**Primary author:** BAŠĒNS, Emīls (Latvian Institute of Organic Synthesis)

**Presenter:** BAŠĒNS, Emīls (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **5** Type: **not specified**

# THE SYNTHESIS OF OCTAHYDROINDOLOQUINOLIZINES VIA IODINE-PROMOTED OXIDATION/BISHLER-NAPIERALSKI CYCLISATION SEQUENCE

*Friday, 17 March 2023 13:20 (20 minutes)*

Abstract

**Primary authors:** ŪDRIS, Niklāvs (Latvian Institute of Organic Synthesis); ŠMITS, Gints

**Presenter:** ŪDRIS, Niklāvs (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **6** Type: **not specified**

# ELECTROCHEMICAL DECARBOXYLATION OF N-SUBSTITUTED 2-AMINOMALONIC ACID MONOESTERS IN INTERMOLECULAR HOFER-MOEST REACTION

*Friday, 17 March 2023 14:40 (20 minutes)*

One of the oldest methods in electroorganic synthesis is Kolbe reaction, where alkyl radical is generated upon anodic decarboxylation. In contrast, Hofer-Moest reaction provides a carbocation after anodic decarboxylation followed by a reaction with a nucleophile.
Aminomalonic acid derivatives are readily available substrates that can be relatively easily functionalized, e.g. by alkylation reactions. Herein we report a previously unreported intramolecular Hofer-Moest reaction of *N*-substituted 2-aminomalonic acid monoesters. A stabilized cation 2 is formed after anodic decarboxylation of a malonic acid monoester 1 followed by intramolecular cyclization. The developed method allows to obtain new tetrahydrofurane and tetrahydropyrane fragment containing amino acid derivatives 3 in good yields.

**Primary authors:** Ms PRANE, Katrina; Ms KOLEDA, Olesja

**Presenter:** Ms PRANE, Katrina

**Session Classification:** Organic chemistry session

Contribution ID: **7** Type: **not specified**

# SYNTHESIS AND USE OF NOVEL MOLECULARLY IMPRINTED POLYMERS FOR SELECTIVE EXTRACTION OF CATECHOLAMINES AND THEIR METABOLITES

*Friday, 17 March 2023 15:20 (20 minutes)*

SYNTHESIS AND USE OF NOVEL MOLECULARLY IMPRINTED POLYMERS FOR SELECTIVE EXTRACTION OF CATECHOLAMINES AND THEIR METABOLITES

JAUNU MOLEKULĀRI IMPRINTĒTU POLIMĒRU SINTĒZE UN TO PIELIETOŠANA SELEKTĪVAI KATEHOLAMĪNU UN TO METABOLĪTU CIETFĀZES EKSTRAKCIJAI

Artūrs Šilaks, Antons Podjava

Laboratory of Chromatography and Mass Spectrometry, Department of Chemistry, Academic Center of Natural Sciences, University of Latvia, Riga, LV-1004, Latvia

e-mail: asilaks@gmail.com

Catecholamines (CAs) are important hormones and neurotransmitters. Abnormal levels of CAs in bodily fluids can be associated with neurodegenerative diseases as well as adrenogenic tumors. Simultaneous determination of CAs and their metabolites in biological fluids is an efficient way of diagnosis and treatment of the aforementioned diseases. Molecularly imprinted polymers (MIPs) are slowly replacing conventional sorbents used in solid-phase extraction (SPE) to achieve superior selectivity for target analyte isolation from complicated matrices. So far there were no attempts to obtain selective sorbents for simultaneous isolation of CAs and their metabolites except the one made by our group [1].

To provide enhanced aqueous stability for polymer particles and improve molecular recognition for both CAs and their metabolites, the MIP is synthesized using methylenebisacrylamide (MBAA, cross-linker, 4) with acrylated homovanillic alcohol (HVAAC, 1), N-(4-vinylbenzyl)-N-methylamine (NVNM, 2) and homovanillic acid (HVA, 3) that act as templates/monomers for CAs and their metabolites, respectively.

MIP sorbents and non-imprinted polymers (NIPs) with varied cross-linker/monomer ratios were prepared and packed into cartridges. Standard analyte mixture was passed through. The imprinting factor (IF), selectivity factor and recovery for each compound were compared to the corresponding NIPs. The preliminary results show that the MIPs have improved retention of CAs compared to NIPs (pH 6). The best-performing MIP will be chosen for further studies.

Table 1. Structural formulas of the compounds chosen for synthesis of the MIPs.

This work is supported by the Fundamental and Applied Research Project lzp-2022/1-0141.

References:

[1] Podjava, A.; Šilaks, A. Synthesis and sorptive properties of molecularly imprinted polymer for simultaneous isolation of catecholamines and their metabolites from biological fluids. J. Liq. Chromatogr. Relat. Technol. 2021, 44, 181–188.

**Primary authors:** PODJAVA, Antons (University of Latvia); ŠILAKS, Artūrs (University of Latvia)

**Presenter:** ŠILAKS, Artūrs (University of Latvia)

**Session Classification:** Organic chemistry session

Contribution ID: **8** Type: **not specified**

# IMPURITY-INDUCED PHOSPHORESCENCE IN CARBAZOLE DERIVATIVES

*Friday, 17 March 2023 15:00 (20 minutes)*

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**Primary authors:** MAZAREVIČS, Artūrs (Latvian Institute of Organic Synthesis); Prof. SUNA, Edgars (Latvian Institute of Organic Synthesis); Dr LEDUSKRASTS, Kaspars (Latvian Institute of Organic Synthesis)

**Presenter:** MAZAREVIČS, Artūrs (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **9** Type: **not specified**

# SYNTHESIS OF LOW-ABUNDANCE SESQUITERPENOIDS FROM β CARYOPHYLLENE

*Friday, 17 March 2023 11:20 (20 minutes)*

β-Caryophyllene is one of the most abundant sesquiterpenes found in nature, therefore it is available at low price from several commercial sources. The unusual structure of β-caryophyllene with two stereodefined chiral centers renders this terpene an attractive renewable starting material for the access of diverse high value compounds.
We demonstrate that β-caryophyllene and its oxide can be used in synthesis of biologically active sesquiterpene lactones rumphellaones A-C [1], disesquiterpenoid rumphellolide J [2], and linariophyllene B (scheme 1). In our ongoing research we show that rare structural units, such as propellane **1**, bridgehead olefins **2a,b** and epoxides **3a,b** (scheme 1) can be prepared from β-caryophyllene in a stereoselective fashion [3]. Such compounds can serve as reference standards for the analysis of constituents of various plant extracts. The biomimetic transformations employed in several chemical steps elucidate the possible biosynthetic route towards natural sesquiterpenoids. Structures of final products were unambiguously confirmed by single crystal X-ray diffraction analysis.

**Primary author:** Mr STAKANOVS, Georgijs (Latvian Institute of Organic Synthesis)

**Co-authors:** Dr RASIŅA, Dace (Latvian Institute of Organic Synthesis); Prof. JIRGENSONS, Aigars (Latvian Institute of Organic Synthesis)

**Presenter:** Mr STAKANOVS, Georgijs (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **10** Type: **not specified**

# SYNTHESIS OF QUINAZOLINES AND INDAZOLES FROM 2-FORMYLPHENYLBORONIC ACIDS

*Friday, 17 March 2023 14:00 (20 minutes)*

Quinazolines and indazoles can be synthesized starting from 2-formylphenylboronic acids using copper-promoted C-N bond formation with subsequent heterocyclization.

**Primary authors:** Ms ZAHAROVA, Darija (Latvian Institute of Organic Synthesis); SOLOMIN, Vitalii

**Presenter:** SOLOMIN, Vitalii

**Session Classification:** Organic chemistry session

Contribution ID: **11** Type: **not specified**

# DESIGN OF S AND SE CONTAINING NUCLEOPHILIC CATALYSTS

*Friday, 17 March 2023 13:00 (20 minutes)*

Pyridine and its derivatives are often used as effective nucleophilic catalysts for reactions such as the Baylis-Hillman reaction, acyl group transfer reactions and others. A noteworthy example is DMAP which is a widely known acylation reaction catalyst. Alcohol acylation reactions can also be catalysed by isochalcogenurea derivatives which exhibit a 1,5-O···Ch interaction in the acylated intermediates [1]. Similar chalcogen bonding interactions haven‘t been investigated in DMAP-type catalysts.

In this research chalcogen containing DMAP-type catalysts were synthesized. Activities of the newly obtained catalysts were determined by performing an acylation reaction of a sterically hindered secondary alcohol (Figure 1). Experiments show that introducing a substituent at the C-2 position significantly decreases the catalytic activity which was expected and has been previously reported [2]. Importantly, it was observed that the activity of sulfur-containing catalysts increases with increasing electron donating ability of the C-4 substituent of pyridine, but the opposite trend was observed for selenium-containing catalysts.

**Primary author:** FILIPSONS, Oto

**Co-author:** KINENS, Artis

**Presenter:** FILIPSONS, Oto

**Session Classification:** Organic chemistry session

Contribution ID: **12** Type: **not specified**

# FLUOROHALOMETHYLSULFONIUM SALTS AS A NOVEL FLUOROHALOCARBENE SOURCE

*Friday, 17 March 2023 10:20 (20 minutes)*

Synthesis of fluorine containing molecules is of great interest due to its unique properties and vast application in pharmaceuticals, agrochemicals and materials [1].
Previously in our group we have developed fluoromethylene transfer from fluromethylsulfonium salts [2, 3]. Herein we wish to report preliminary results on synthesis of reagents 1 and its initial application in carbene transfer reaction (Scheme 1).

Scheme 1. Alkene cyclopropanation with fluorohalomethylsulfonium salts.
We have found that functionalized sulfonium salts – fluorohalomethylsulfonium reagents 1 are efficient source of fluorhalocarbene under basic conditions and they undergo unactivated alkene 2 cyclopropanation to deliver fluorohalocyclopropanes 3.

References:
[1] Zhou, Y., et al. Chem. Rev. 2016, 116, 422–518.
[2] Melngaile, R., Veliks, J. Synthesis 2021, 53, 4549-4558.
[3] Sperga, A., Zacs, D., Veliks J. Org. Lett. 2022, 24, 4474–4478.

**Primary author:** SPERGA, Artūrs (Latvian Institute of Organic Synthesis)

**Co-author:** VELIKS, Jānis

**Presenter:** SPERGA, Artūrs (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **13** Type: **not specified**

# C-H ARYLATION OF PENTACYCLIC TRITERPENOIDS

*Friday, 17 March 2023 10:40 (20 minutes)*

C-H activation of betulin, oleanolic acid and ursolic acid

**Primary author:** KROŠKINS, Vladislavs (RTU, OĶTI.)

**Presenter:** KROŠKINS, Vladislavs (RTU, OĶTI.)

**Session Classification:** Organic chemistry session

Contribution ID: **14** Type: **not specified**

# ACRAB-TOLC EFFLUX PUMP INHIBITOR ANALOG SYNTHESIS

*Friday, 17 March 2023 12:40 (20 minutes)*

Laura Pauniņa1, Cristina Durante Cruz2, Marina Madre1, Tania Szal3, Päivi Tammela2, Aigars Jirgensons1, Björn Windshügel3, Jānis Veliks1
1 Latvian Institute of Organic Synthesis, Aizkraukles 21, LV-1006, Riga, Latvia
2 Drug Research Program, Division of Pharmaceutical Biosciences, Faculty of Pharmacy, University of Helsinki, Finland P.O. Box 56 (Viikinkaari 5E), FI-00014, Helsinki, Finland
3 Discovery Research ScreeningPort, Fraunhofer Institute for Translational Medicine and Pharmacology ITMP, Schnackenburgallee 114, 22525 Hamburg, Germany
e-mail: laura.paunina@osi.lv

Bacterial resistance to the existing classes of antibiotics is one of the most important challenges for the future healthcare system and bacterial cells efflux pumps play an important role for this internal drug resistance. To reduce the ability of the efflux pumps binding to medication substrates, the molecules called efflux pump inhibitors are used to rejuvenate the antibiotics activity by binding to the efflux pump protein [1].
In the framework of the project, it was hypothesized that AcrAB-TolC efflux pump outer membrane protein TolC in Gram-negative E.coli bacteria cells could represent an attractive drug target. Therefore, structure analogs of known clinical candidate compound have been synthesized and identified their structure-activity relationships (SAR) to TolC efflux pump [2].

Acknowledgements
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References:

[1] AlMatar. M., Albarri. O., Makky. E. A., Köksa. F. Pharmacol Rep., 2021, 73, 1-16.
[2] Ott. G. R., Cheng. M., Learn. K. S., Wagner. J., Gingrich. D. E., Lisko. J. G., Dorsey. B. D. Journal of Medicinal Chemistry, 2016, 59(16), 7478-7496.

**Primary authors:** PAUNIŅA, Laura; Dr DURANTE CRUZ, Cristina; Dr MADRE, Marina; SZAL, Tania; Prof. TAMMELA, Päivi; Prof. JIRGENSONS, Aigars; Prof. WINDSHÜGEL, Björn; Dr VELIKS, Jānis

**Presenter:** PAUNIŅA, Laura

**Session Classification:** Organic chemistry session

Contribution ID: **15** Type: **not specified**

# AZIDE-TETRAZOLE EQUILIBRIUM IN PYRIDO[3,2-d]PYRIMIDINES

*Friday, 17 March 2023 10:00 (20 minutes)*

Heterocycles with an azido-azomethine structural entity are interesting due to their intrinsic dynamic azide-tetrazole tautomeric equilibrium in the solution phase [1] alongside rich azide functional group chemistry [2].
Herein, a method for the synthesis of 5-substituted tetrazolo[1,5-a]pyrido[2,3 e] pyrimidines from 2,4-diazidopyrido[3,2-d]pyrimidine in SNAr reactions with N-, O-, and S nucleophiles is presented [3]. The tetrazolo[1,5-a]pyrimidine derivatives can be regarded as 2 azidopyrimidines due to present azide-tetrazole valance tautomerism and functionalized in copper(I)-catalyzed azide-alkyne dipolar cycloaddition (CuAAC) and Staudinger reactions.
Equilibrium constants and thermodynamic values were determined using variable temperature 1H NMR and were found to be ΔG298 = −3.33 to −7.52 (kJ/mol), ΔH = −19.92 to −48.02 (kJ/mol) and ΔS = −43.74 to −143.27 (J/mol·K). The negative Gibbs free energy values assert tetrazole as the major tautomeric form in solutions. Furthermore, the key starting material 2,4-diazidopyrido[3,2-d]pyrimidine shows a high degree of tautomerization in different solvents presenting up to 7 tautomeric forms.

**Primary authors:** LEŠKOVSKIS, Kristaps (Riga Technical University); Dr NOVOSJOLOVA, Irina; Prof. TURKS, Māris

**Presenter:** LEŠKOVSKIS, Kristaps (Riga Technical University)

**Session Classification:** Organic chemistry session

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# DEVELOPMENT OF BENZOXAPHOSPHEPINE 2-OXIDES AS CARBONIC ANHYDRASE INHIBITORS

*Friday, 17 March 2023 14:20 (20 minutes)*

Carbonic anhydrases (CA, EC 4.2.1.1) are essential metalloenzymes found across all kingdoms of life. These enzymes are involved in many important physiological processes, as they catalyse the reversible hydration of carbon dioxide [1]. To date, 15 different human CA isoforms have been identified, out of which CA IX and XII isoforms are highly overexpressed in different tumour types and may contribute to the progression of cancer [2]. Therefore, there is a particular need to develop potent and selective CA inhibitors.

Herein we report our results on the development of a new class of CA inhibitors — benzoxaphosphepine 2-oxides. Aforementioned compounds showed a remarkable selectivity and good activity against the tumour-associated isoforms CA IX and XII [3]. Furthermore, these compounds can be used as starting points for the design of more potent CA IX/XII inhibitors.

**Primary author:** Ms BALAŠOVA, Anastasija (Latvian Institute of Organic Synthesis)

**Co-author:** Dr ŽALUBOVSKIS, Raivis (Latvian Institute of Organic Synthesis)

**Presenter:** Ms BALAŠOVA, Anastasija (Latvian Institute of Organic Synthesis)

**Session Classification:** Organic chemistry session

Contribution ID: **17** Type: **not specified**

# SYNTHESIS OF POTENTIAL IRE1α INHIBITORS

*Friday, 17 March 2023 13:40 (20 minutes)*

Cancer has a major impact on society around the world and it is one of the leading causes of death. IRE1α is an enzyme that plays a part in the development of certain cancers, such as breast cancer, colon cancer, and prostate cancer. IRE1α inhibitors might be used to treat these types of cancer. [1]
The aim of this study was to find IRE1α inhibitors that would have a greater selectivity and bioavailability than the previously discovered ones. Based on computational data about the activity of compound 4f, it was chosen as the model compound for further synthesis.
The reaction used for the synthesis of compound 4f and its analogues was found to yield endocyclically acylated 1,2,4-triazol-5-amines instead of the anticipated exocyclically acylated compounds, but isomerization of endocyclically acylated 1,2,4-triazol-5-amines yielded exocyclically acylated compounds. However, the limited hydrolytic stability of compounds 3a-f suggested that the inhibitory activity of these compounds could be mainly due to the presence of compounds 2a-f in solution. Indeed, compound 2f had the greatest ability to inhibit IRE1α out of all the synthesized compounds.

**Primary author:** ŠADAUSKIS, Jānis

**Co-authors:** Dr KĻIMENKOVS, Igors; Prof. SŪNA, Edgars

**Presenter:** ŠADAUSKIS, Jānis

**Session Classification:** Organic chemistry session

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# USE OF PROPARGYLSILANES FOR THE PREPARATION OF HIGHLY FUNCTIONALIZED ALKENES VIA 1,2-SILYL MIGRATION

*Friday, 17 March 2023 09:40 (20 minutes)*

The ease of the unsaturated system reactivity proceeding via □-silyl carbocation ion can be explained by the stabilizing effects of the silicon-carbon bond interaction with carbocation ion - known as □-silicon effect. This can be achieved by either vertical (hyperconjugation) or non-vertical (formation of cyclic silonium ion) stabilization. The formation of the latter, in combination with other stabilizing effects, causes 1,2-silyl migration [1].
Previously, we have reported the use of Brønsted acid to catalyze reactions of propargyl silanes to form various silyl dienes and indenes [2,3]. Herein, we report the expanded use of the concept by using electrophilic bromine to induce the formation of the reactive allylic cation that readily reacts with a variety of nucleophilic solvents like methanol, dimethylformamide, and acetic acid to form allyl functionalized vinyl silanes.
Use and the functionality of the obtained vinyl silanes are showcased in a variety of transition metal-catalyzed transformations like Suzuki-Miyaura coupling, C-H activation, electrophilic silicon exchange reaction, and Lewis acid-promoted intramolecular cyclization to form indenes.

**Primary authors:** Mr PURIŅŠ, Mikus (Riga Technical University); BEĻAUNIEKS, Rūdolfs (Riga Technical University); Prof. TURKS, Māris (Riga Technical University); LĪPIŅA, Rebeka Anna (Riga Technical University)

**Presenter:** BEĻAUNIEKS, Rūdolfs (Riga Technical University)

**Session Classification:** Organic chemistry session

Contribution ID: **19** Type: **not specified**

# USE OF TERMINALLY FUNCTIONALIZED PROPARGYL SILANES FOR THE SYNTHESIS OF VARIOUS 5-MEMBERED HETEROCYCLES VIA 1,2-SILYL MIGRATION

*Friday, 17 March 2023 09:20 (20 minutes)*

Small heterocycles, particularly those containing a 5-membered cycle, are popular motifs in pharmaceuticals, displaying a broad range of biological properties [1]. A well-established strategy for the synthesis of 5-membered saturated/partially saturated heterocycles involves intramolecular cyclization, made possible by internal nucleophile attack on carbocations.
In this work we investigate the use of electrophile induced 1,2-silyl migration in terminally functionalized propargyl silanes to generate stabilized carbocations, capable of reacting with various internal nucleophiles, forming heterocyclic units (scheme 1). Various nucleophilic species could be utilized, namely alcohols, carboxylic acids, oximes, acyl and sulfonyl amides, carbamates and thioacetates.
The synthetic utility of the cyclization products was demonstrated by difunctionalization of the alkene moiety in cross-coupling reactions to selectively obtain trisubstituted alkenes. The resulting heterocycle derivatives were obtained with a high degree of stereoselectivity and yields up to 82%.

References:
[1] Vitaku, E.; Smith, D. T.; Njardarson, J. T. J. Med. Chem. 2014, 57, 10257–10274.

**Primary author:** KROŅKALNE, Rasma (RTU)

**Co-authors:** Mr BEĻAUNIEKS, Rūdolfs (RTU); Mr UBAIDULLAJEVS, Artjoms (RTU); Prof. TURKS, Māris (RTU)

**Presenter:** KROŅKALNE, Rasma (RTU)

**Session Classification:** Organic chemistry session